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SUPERSOLIDUS SINTERING AND MECHANICAL PROPERTIES OF WATER-ATOMISED M3/2 HIGH-SPEED STEEL POWDER SINTERED UNDER NITROGEN-BASED ATMOSPHERE

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Abstract. The ability to produce near net shape high-speed steels using a continuous belt furnace offers the opportunity to improve the properties and reduce the processing cost of these materials. So far, the difficulty in making this advancement has been the requirement of high sintering temperatures (1250–1350°C) in vacuum atmosphere. A low cost-sintering route of water-atomised high-speed steel powder has been developed during this project using Supersolidus Liquid Phase Sintering (SLPS) mechanism. This water-atomised M3/2 high-speed steel powder can be sintered to full density (>95% of theoretical density) under nitrogen-based atmosphere at sintering temperature of 1150°C. Acceptable microstructures and mechanical properties have been obtained after appropriate heat treatment.

Keywords: Supersolidus, water-atomised powder, nitrogen-based atmosphere

Abstrak. Keupayaan untuk menghasilkan keluli laju-tinggi kepada bentuk hampir sebenar menggunakan relau tali-sawat berterusan menawarkan peluang untuk mempertingkatkan sifat-sifat serta mengurangkan kos pemprosesan bahan keluli ini. Sehingga kini, masalah yang timbul bagi mencapai matlamat tersebut adalah keperluan menggunakan suhu persinteran tinggi (1250–1350°C) di dalam atmosfera vakum. Satu kaedah persinteran yang murah terhadap serbuk keluli laju-tinggi pengabusan-air telah dibangunkan menerusi projek ini menggunakan mekanisme Persinteran Fasa Pepejalan-lampau Cecair (PFPC). Serbuk M3/2 keluli laju-tinggi ini boleh tersinter kepada ketumpatan penuh (>95% daripada ketumpatan teori) di dalam atmosfera berasaskan-nitrogen pada suhu persinteran 1150°C. Mikrostruktur dan sifat-sifat mekanik yang baik telah berjaya dihasilkan selepas proses rawatan haba yang sesuai dilakukan.

Kata kunci: Pepejalan-lampau, serbuk pengabusan-air, atmosfera berasaskan-nitrogen

1.0 INTRODUCTION

In order to satisfy the requirements from the metal cutting industry for better tolerances and manufacturing cost savings, several new powder metallurgy (PM) processing routes for the production of high-speed steels (HSS) have been developed in recent decades [1]. These PM production routes conventionally can be categorised into two main groups: the manufacturing of bars or billets by hot isostatic pressing (HIP) of prealloyed gas-atomised powders; and the production of near net shape parts by

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cold pressing and sintering of prealloyed water-atomised powders. A third, relatively new technique, is the production of high complexity near net shape part by powder injection moulding (PIM). However, these three techniques need high sintering temperatures (1250–1350°C), and require sintering in vacuum atmosphere (generally no better than ± 3°C).

An alternative processing route has been developed, which not only promises lower manufacturing costs of HSS tools, but also offers additional benefit in terms of tool life and surface finish, compared with other methods [1]. Recently, sintering of HSS powder containing high vanadium under nitrogen-based atmosphere [2], and the addition of phosphorus to HSS powder, followed by sintering under vacuum atmosphere [3] have proven to be an effective method of lowering the sintering temperature to a level at which, components can be sintered to full density at optimum sintering temperature (OST) of 1150°C. In the present work, phosphorus element was introduced by blending iron-phosphorus powders into the HSS powder before sintering in furnace under nitrogen-based atmosphere condition.

The supersolidus liquid phase sintering (SLPS) concepts [4] shown in Figure 1 is a process involving heating prealloyed powders to a point intermediate between the solidus and liquidus, promoting partial melting and densification through the operation of particle arrangement has been followed. Powder will sinter to full density over a range of temperatures until the upper limit of this range is reached, i.e. when specimens show distortion, defined by rounding of corners or general loss of shape. In reality, the practical limit is reached before distortion, though for any alloy grade there is a narrow temperature band – *the sintering window* – which will produce acceptable microstructures and properties [5]. The sintering window concept is illustrated

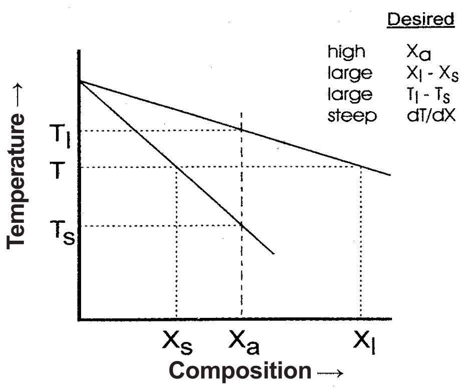


Figure 1 A schematic phase diagram plot showing the desired features for super-solidus liquid phase sintering (SLPS) mechanism [4]

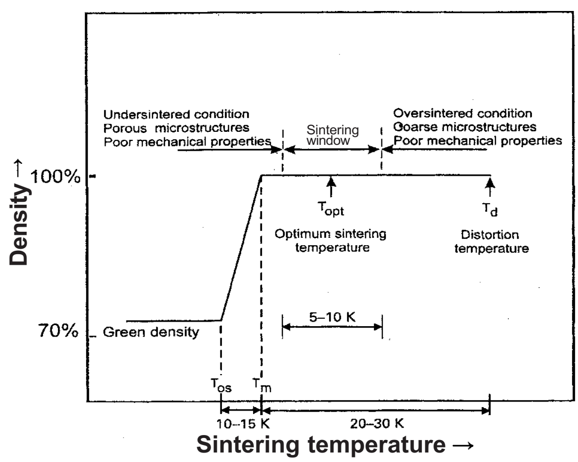


Figure 2 Schematic diagram showing the relationship between the optimum sintering temperature and sintering window for prealloyed HSS powders in vacuum atmosphere [5]

schematically in Figure 2. Thus a major factor determining the successful processing of water atomised HSS powders to full density is the width of the sintering window, the temperature interval in which sintering must take place if optimum microstructures and mechanical properties are to be achieved. Therefore, it is the aim of this work not only to reduce the sintering temperature but also widen the sintering window range of M3/2 high-speed steel powder.

2.0 MATERIALS AND METHODS

Sintering trials were carried out on an-annealed water-atomised M3 class 2 (M3/2) HSS powder of mean particle size 75 μm with the following composition (wt-%): 1.01C, 4.04Cr, 5.71Mo, 5.96W, 3.25V, 0.39Co, and 79.64Fe. Phosphorus addition was introduced into the HSS powder by dry mixing in a double cone mixer with 5, 7, and 9wt-% of iron-phosphorus (Fe_3P) powder.

Green compacts, 15 × 15 mm, 5 mm thick, were die pressed without lubricant at 11 tons (800 MPa) to attain ~75% of theoretical density. These green bodies were heated to the sintering temperature at a heating rate of 10°C/min under nitrogen-based gases (95%N_2+5%H_2) and furnace cooled to room temperature, after holding for 1 hour. The densities of the sintered specimens were determined by Archimedes method. The heat treatment process used for hardening this HSS consists of austenitising at 1050°C in argon atmosphere for 5 minutes, followed by an oil quenching. A triple tempering was carried out at temperatures between 500 – 600°C, each tempering last for 1 hour, followed by intermediate cooling.

Samples for the metallographic examination were prepared using standard techniques and etched in 5% nital. The specimens were metallographically examined using optical, and scanning electron microscope (SEM). The identification of the phases in the microstructures was determined using energy dispersive X-ray spectrometer (EDAX). Hardness measurements were performed using a 100 g load on sectioned and polished specimens (both in the as quenched and in the tempered state). Three point bend tests were used to determine the transverse rupture strength (TRS) in selected specimens that were considered to have approached their optimum heat treatment properties. Tests were carried out on span L= 10 mm, width W= 2 mm, and depth D= 4 mm cut from the heat treated samples, and polished to a 1 μm diamond finish on the tensile face. Values of TRS are given by [6]:

$$TRS = \frac{3PL}{2WD^2} \qquad \text{where } P \text{ is the load}$$

3.0 RESULTS AND DISCUSSION

Sintering curves obtained for the M3/2 HSS powder mixed with iron-phosphorus alloy are shown in Figure 3. The curves illustrate the effects of addition of Fe_3P

powders, and show the variations of sintering temperature with sintered density. As the addition of Fe_3P powder increased to 9 wt-%, the sintering temperature required to achieve near full density (>95% of TD) was reduced to 1150°C, compared with 1325°C for M3/2 HSS powder. For the alloys of lower Fe_3P content (5 and 7wt-%), the optimum sintering temperatures (OST) occurred at 1200 and 1175°C, respectively.

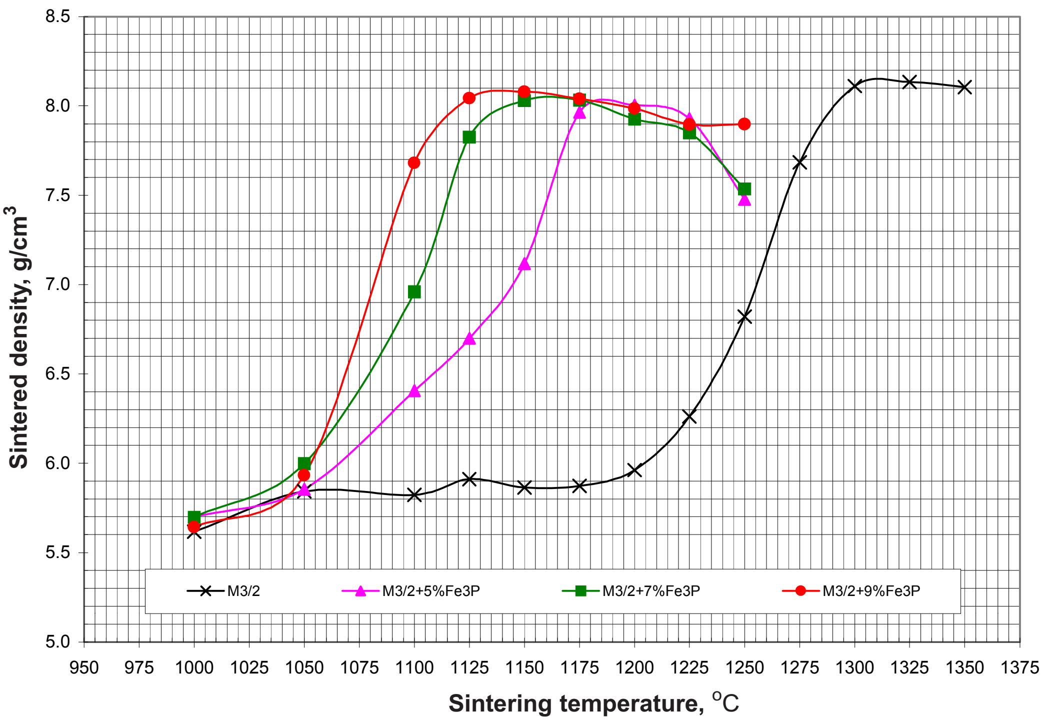
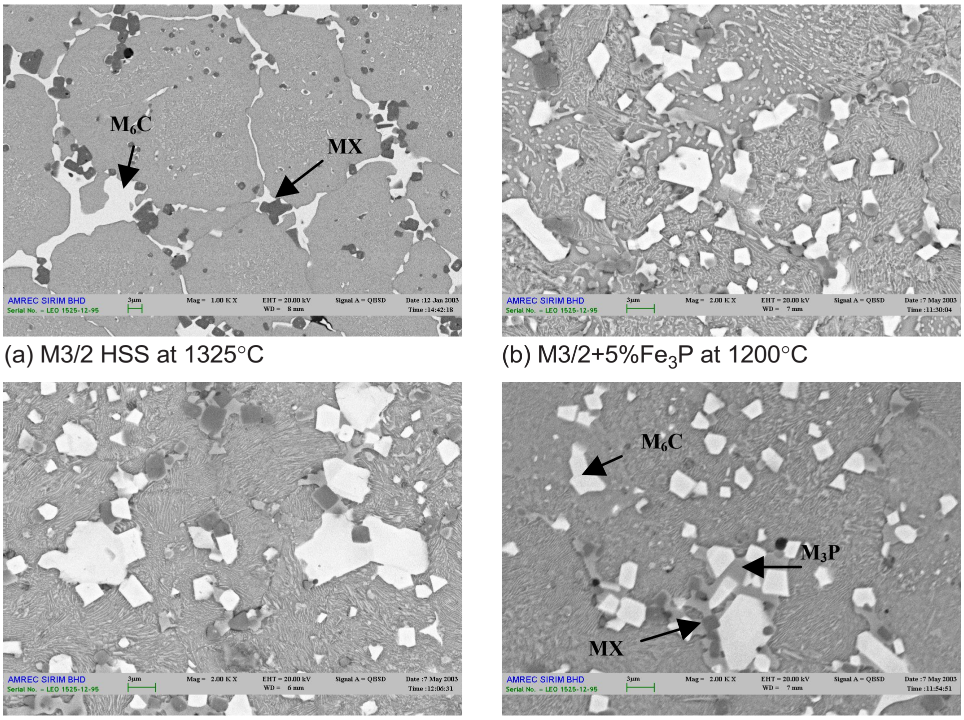


Figure 3 Sintering curves of M3/2 HSS and its alloy

The densification of HSS process with Fe_3P addition is more rapid at 1050°C, compared to 1200°C for M3/2 HSS powder. From this study, it was also found that the addition of Fe_3P in M3/2 HSS powder not only decrease the optimum sintering temperature but also widen the sintering window range up to ± 50°C, similar to work done by another research [3]. Therefore at this point, the possibility of sintering M3/2 HSS powder in industrial continuous belt furnace is significant and could meet the need of powder metallurgy industries.

In the 'as-sintered' condition, the microstructures of M3/2 HSS samples comprise of large size Fe-Mo-W rich M_6C, and globular vanadium-rich MX carbonitrides as shown in Figure 4(a). For alloys with Fe_3P addition, the microstructures consist of angular M_6C carbide, finer MX carbonitrides, and M_3P type phosphides (Figure 4(b-d)). Typical compositions of these phases are shown in Table 1. At the distortion temperature T_d, 1250°C, the 'herringbone' of M_3P, eutectic structure of M_6C, and



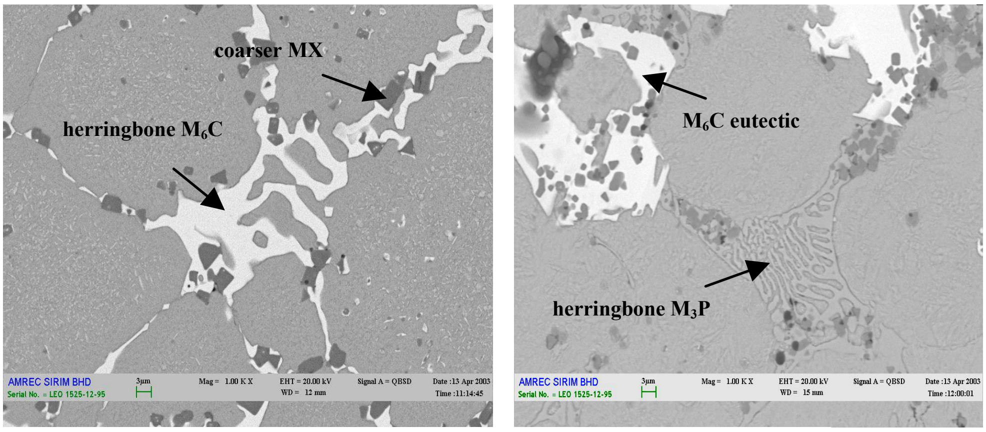
(a) M3/2 HSS at 1325°C
(b) M3/2+5%Fe_3P at 1200°C
(c) M3/2+7%Fe_3P at 1175°C
(d) M3/2+9%Fe_3P at 1150°C

Figure 4 Scanning electron micrographs (SEM) of samples sintered at OST

coarser size of MX carbonitrides were observed within the microstructure of distorted samples (Figure 5(b). Therefore, it is a good practice to sinter these alloys below distortion temperature in order to avoid the eutectic structure and shape changes.

Table 1 EDAX analysis of M3/2 HSS sintered at OST

	Composition, ωt-%						
Phase	P	V	Cr	Fe	Co	Mo	W
M3/2 HSS (OST = 1325°C)							
M_6C	-	6.85	7.65	37.93	7.29	21.24	19.04
MX	-	90.06	-	5.04	2.29	1.50	1.11
M3/2+9%Fe_3P (OST = 1150°C)							
M_6C	3.74	1.03	8.06	49.29	0.53	20.63	16.72
M_3P	19.81	8.25	15.89	46.70	3.04	5.83	0.49
MX	3.84	59.21	7.40	24.64	0.15	3.14	1.62



(a) M3/2 at 1350°C (b) M3/2+9%Fe_3P at 1250°C

Figure 5 SEM micrographs of oversintered samples

In order to obtain maximum benefit in relation to hardness and temper resistance, it is necessary to heat treat (austenitising and quenching) sintered specimens very close to the solidus temperature. Generally, HSS is austenitised at 30-40°C below the solidus [7]. In this study, sintered specimens of M3/2+9%Fe_3P were austenitised at 1050°C. Hardness was measured after quenching and triple tempering. The peak hardness of 890 HV1.0 was achieved at a tempering temperature of 525°C (Figure 6). The results of three-point bend test are given in Table 2. The hardness and bend strength values obtained in this work are comparable with other PM high-speed steel [3], as shown in Table 3.

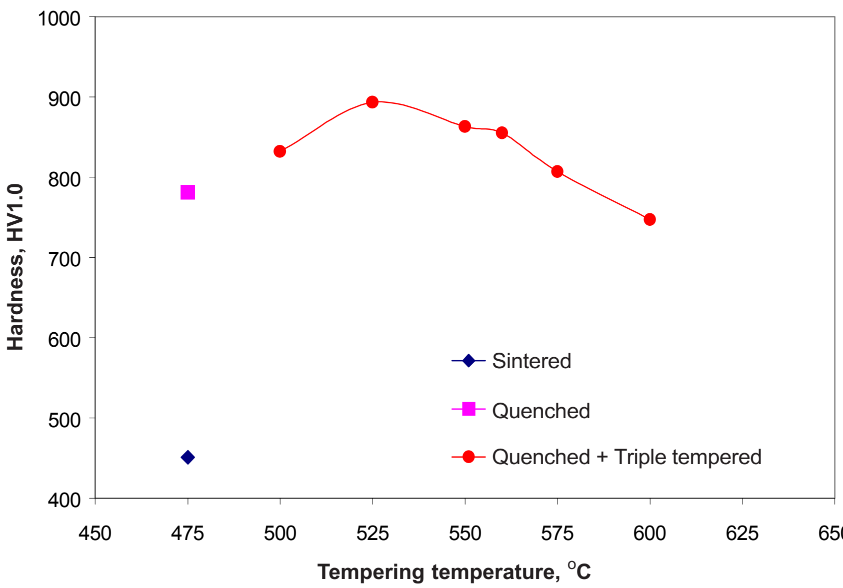


Figure 6 Effect of tempering temperature on hardness of M3/2+9%Fe_3P austenitised at 1050°C

Table 2 Average TRS of hardened and tempered specimen of M3/2+9%Fe_3P, austenitised at 1050°C

Tempering temperature, °C	Hardness, HV1.0	TRS, GPa
500	832	1.06
525	893	1.15
550	863	1.31
575	807	1.24

Table 3 Mechanical properties (average values) of PM HSS

Grade	Hardness, HV	TRS, GPa
M2 [3]	890	1.2
T1 [3]	870	1.3
ASP60 [5]	955-963	1.5-2.0
* M3/2	863-893	1.2-1.3

* Present work

4.0 CONCLUSIONS

From this work, it has been demonstrated that M3/2 HSS powder can be sintered to full density at temperatures as low as 1150°C, and it is possible to be sintered using an industrial continuous belt furnace. The additions of 5 and 7 wt-% Fe_3P into M3/2 HSS powder produced fully dense microstructures after nitrogen-based atmosphere sintering at sintering temperatures of 1200 and 1175°C, respectively. However, the addition of 9 wt-% Fe_3P into this powder, not only reduces the sintering temperature to 1150°C but also widens the sintering window to 75°C. Acceptable microstructures and good mechanical properties were also obtained during this work, which are comparable with other Powder Metallurgy (PM) HSS powders.

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